



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3085

Aroclor 1254 in Methanol

This Standard Reference Material (SRM) is a solution of Aroclor 1254 (Chemical Abstracts Registry Number 11097-69-1) in methanol. This SRM is intended primarily for calibrating chromatographic instrumentation and methods of analysis used for the determination of Aroclor 1254 and polychlorinated biphenyls (PCBs) in water. A unit of SRM 3085 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Aroclor 1254: The certified concentration [1,2], given below, is based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Aroclor 1254: 7.08 mg/kg \pm 0.16 mg/kg or 5.66 mg/L \pm 0.13 mg/L

The result is expressed as the certified value \pm the expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and gas chromatographic measurements. The expanded uncertainty, at the 95 % level of confidence, is calculated as $U = ku_c$, where u_c is a combined standard uncertainty calculated according to the ISO and NIST Guides [3] and $k = 2$ is the coverage factor. The value of u_c includes an allowance for differences between the concentration determined by gas chromatographic measurements for various sources of Aroclors and gravimetric preparation. The concentration expressed in mg/L was obtained by multiplying the certified value, expressed as a mass fraction, by the measured density (22 °C) of the SRM solution, 0.7996 g/mL \pm 0.0153 g/mL, where 0.0153 represents one standard deviation (1s) and is incorporated in the uncertainty associated with the concentration expressed in mg/L.

Expiration of Certification: The certification of this SRM lot is valid until **31 July 2012**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification was under the direction of D.L. Poster and M.M. Schantz of the NIST Analytical Chemistry Division.

Analytical measurements of the SRM were performed by D.L. Poster of the NIST Analytical Chemistry Division.

Partial support for the preparation and certification of this Standard Reference Material was provided by the U.S. Environmental Protection Agency Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

Stephen A Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 15 July 2005
See Certificate Revision History on Last Page

Preparation of the SRM was performed by M.P. Cronise of the NIST Measurement Services Division and D.L. Poster of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. McDonald of the NIST Measurement Services Division.

NOTICE AND WARNING TO USERS

Handling: This material is a solution of a PCB mixture. PCB-containing materials are reported to be toxic. Extreme caution and care should be exercised during the handling of SRM 3085. Contact your regional EPA office for information regarding proper disposal.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

INSTRUCTIONS FOR USE

Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified value to be valid within the stated uncertainty. Because of the volatility of methanol, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS¹

The Aroclor 1254 used in the preparation of this SRM was obtained from the U.S. EPA. The SRM was prepared at NIST by weighing and mixing Aroclor 1254 into methanol. The Aroclor was added to the methanol and mixed until completely dissolved and homogenized. The total mass of this solution was measured and aliquots (1.2 mL) were dispensed into 2-mL amber glass ampoules, which were then flame sealed.

Aliquots from two sets of nine and five ampoules, respectively, selected randomly, were analyzed using capillary gas chromatography with electron capture detection and an immobilized non-polar (5 % phenyl methylpolysiloxane) stationary phase column. An internal standard solution containing 2,4,5-trichlorobiphenyl and 2,2',4,6,6'-pentachlorobiphenyl was added to the first set of samples for quantification purposes. Calibration solutions consisting of weighed amounts of Aroclor 1254 and the internal standard compounds in methanol were chromatographically analyzed to determine response factors for Aroclor 1254 relative to each internal standard. An internal standard solution containing 2,4,6-trichlorobiphenyl and 2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl was added to the second set of samples for quantification purposes. Calibration solutions consisting of weighed amounts of Aroclor 1254 from five different sources and the internal standard compounds in methanol were chromatographically analyzed to determine response factors for Aroclor 1254 relative to each internal standard. The results for Aroclor 1254 are based on the areas of the dominant Aroclor PCB peaks relative to the internal standard peaks (Figure 1). This approach is similar to U.S. EPA Method 505 (Analysis of organohalide pesticides and commercial polychlorinated biphenyl [PCB] products in water by microextraction and gas chromatography, revision 2.0).

¹Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

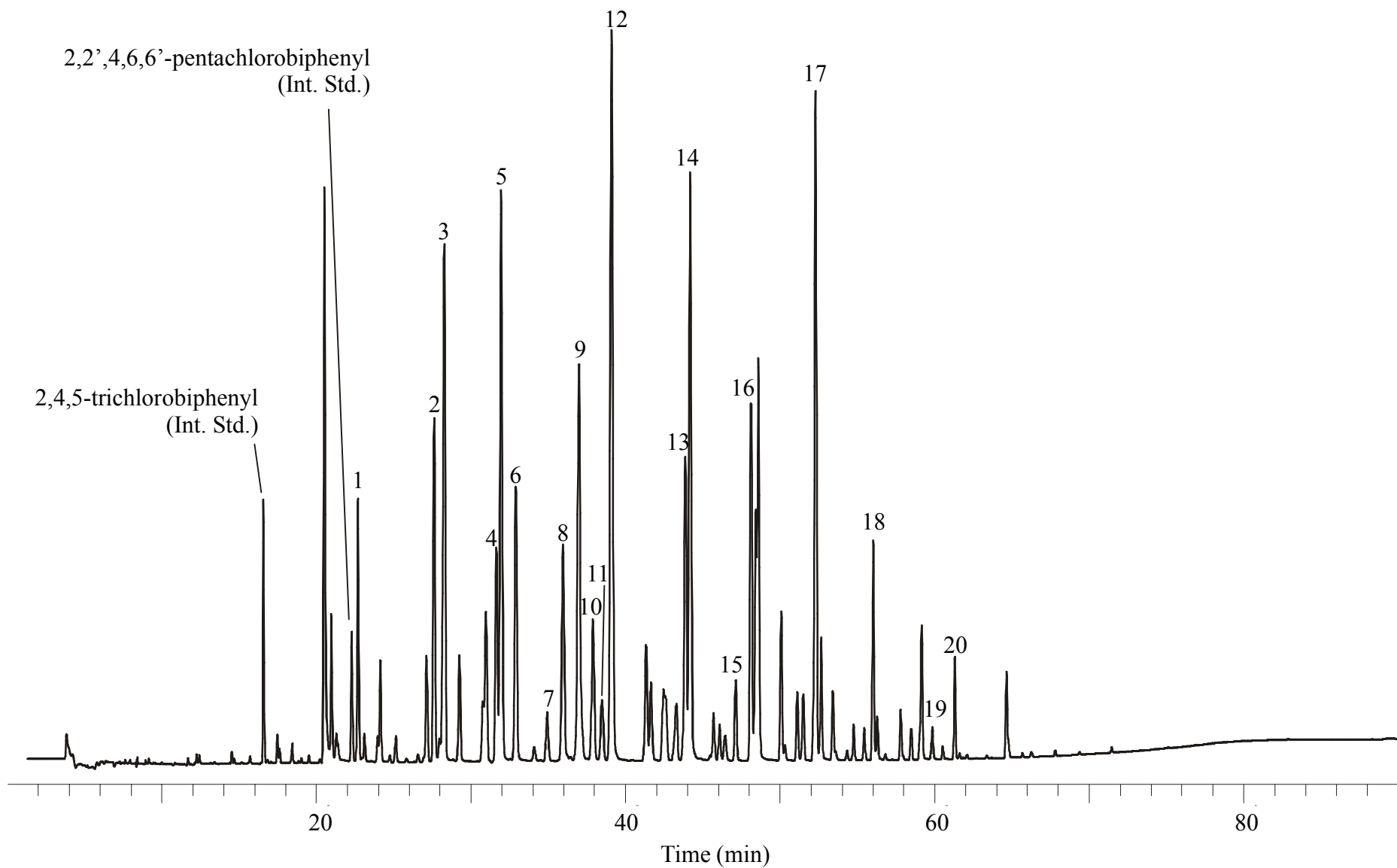


Figure 1. Gas chromatogram from the analysis of SRM 3085 (Aroclor 1254 in Methanol). The 20 peaks used for quantification of the Aroclor mass in solution are shown. (Int. Std.) = internal standard

REFERENCES

- [1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).
- [2] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (1995).
- [3] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.

Certificate Revision History: 15 July 2005 (This technical revision clarifies the determination of the certified value expressed in mg/L); 23 May 2003 (Original certificate date).
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Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.